

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Patricia Preikschat et al.

Title: CHROMIUM(VI)-FREE CONVERSION LAYER AND METHOD  
FOR PRODUCING IT

Attorney  
Docket No.: 31716US1

FIRST PRELIMINARY AMENDMENT  
(Submitted With Initial Filing)

Commissioner for Patents  
Washington, D.C. 20231

Dear Sir:

Please amend the above-referenced patent application as follows prior to examination.

IN THE SPECIFICATION:

On page 1, line 5 of the specification, immediately below the title, please insert the following section and accompanying paragraph:

CROSS-REFERENCE TO RELATED APPLICATIONS

This application is a continuation of U.S. Patent Application Serial No. 09/171,558 filed on March 29, 1999, which is a § 371 of PCT Application Serial No. PCT/DE97/00800 filed April 18, 1997.

At the top of page 24 above claim 1, please delete "Claims" and insert the following starting from the left margin of the page:

WHAT IS CLAIMED IS:

I hereby certify that this being deposited with the U.S. Postal Service as Express Mail, Post-Office to Addressee, Express Mailing Label No. EL653124184US addressed to: Commissioner for Patents, Washington, D.C. 20231 on the date indicated below.

John P. Murtaugh  
Name of Attorney for Applicant  
7-13-01 John P. Murtaugh  
Date Signature of Attorney

IN THE CLAIMS:

Please amend claims 3-6, 9, 12, 13, 16-20, 22, 24, and 26-28 as follows.

3. (amended) A conversion layer according to claim 1, characterised in that it may contain, for further enhanced corrosion protection, additional components selected from the group consisting of: silicate, cerium, aluminum and borate;

additional metal compounds, in particular 1- to 6-valent metal compounds, for example compounds of Na, Ag, Al, Co, Ni, Fe, Ga, In, lanthanides, Zr; Sc, Ti, V, Cr, Mn, Cu, Zn, Y, Nb, Mo, Hf, Ta, W; and

anions, in particular halide ions, in particular chloride ions; sulfurous ions, in particular sulfate ions, nitrate ions; phosphoric ions, in particular phosphate ions, diphosphate ions, linear and/or cyclic oligophosphate ions, linear and/or cyclic polyphosphate ions, hydrogen phosphate ions; carboxylic acid anions; and silicon-containing anions, in particular silicate anions; and

polymers, in particular organic polymers, corrosion inhibitors; silicic acids, in particular colloidal or disperse silicic acids; surfactants; diols, triols, polyols; organic acids, in particular monocarboxylic acids; amines; plastics dispersions; dyes, pigments, in particular carbon black, chromogenic agents, in particular metallic chromogenic agents; amino acids, in particular glycine; siccatives, in particular cobalt siccatives; dispersing agents; and

mixtures thereof.

4. (amended) A conversion layer according to claim 1, characterised in that it is a basis for further inorganic and/or organic layers.

5. (amended) A conversion layer according to claim 1, characterised in that it contains dyes or color pigments for modification of the inherent color thereof.

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6. (amended) A conversion layer according to claim 1, characterised in that its layer thickness is approx. 100 nm.
9. (amended) A method according to claim 7, characterised in that the ligands of the chromium(III) complex are selected from the group consisting of:
  - chelate ligands, such as dicarboxylic acids, tricarboxylic acids, hydroxycarboxylic acids, in particular oxalic, malonic, succinic, glutaric, adipic, pimelic, suberic, azelaic, sebacic acid; and
  - furthermore, maleic acid, phthalic acid, terephthalic acid, tartaric acid, citric acid, malic acid, ascorbic acid; and
  - Further chelate ligands such as acetylacetone, urea, urea derivatives, and
  - further complex ligands wherein the complexing functional group contains nitrogen, phosphorus or sulfur (-NR<sub>2</sub>, -PR<sub>2</sub>, wherein R independently is an organic, in particular aliphatic radical and/or H, and/or -SR, wherein R is an organic, in particular aliphatic radical or H); phosphinates and phosphinate derivatives; as well as
  - suitable mixtures thereof, among each other as well as in mixed complexes with inorganic anions and H<sub>2</sub>O and/or
- the method is performed repeatedly on the surface to be passivated.

12. (amended) A concentrate according to claim 10, characterised in that the concentrate is present in solid or liquid form.

13. (amended) A concentrate according to claim 10,  
characterised in that it contains further additives selected from  
the group consisting of: sealers, dewatering fluids; and  
  
additional metal compounds, in particular 1- to 6-valent metal  
compounds, for example compounds of Na, Ag, Al, Co, Ni, Fe,  
Ga, In, lanthanides, Zr; Sc, Ti, V, Cr, Mn, Cu, Zn, Y, Nb, Mo,  
Hf, Ta, W; and  
  
anions, in particular halide ions, in particular chloride ions;  
sulfurous ions, in particular sulfate ions, nitrate ions;  
phosphoric ions, in particular phosphate ions, diphosphate  
ions, linear and/or cyclic oligophosphate ions, linear and/or  
cyclic polyphosphate ions, hydrogen phosphate ions;  
carboxylic acid anions; and silicon-containing anions, in  
particular silicate anions; and  
  
polymers, in particular organic polymers, corrosion inhibitors;  
silicic acids, in particular colloidal or disperse silicic acids;  
surfactants; diols, triols, polyols; organic acids, in particular  
monocarboxylic acids; amines; plastics dispersions; dyes,  
pigments, in particular carbon black, chromogenic agents, in  
particular metallic chromogenic agents; amino acids, in  
particular glycine; siccatives, in particular cobalt siccatives;  
dispersing agents; as well as  
  
mixtures thereof.

16. (amended) A passivation bath according to claim 14,  
in that it has a pH between approx. 1.5 and 3.

17. (amended) A passivation bath according to claim 14,  
characterised in that it contains approx. 20 g/l chromium(III)  
and has a pH of approx. 2 to 2.5.

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18. (amended) A passivation bath according to claim 14, characterised in that it contains further additives in particular selected from the group consisting of sealers, dewatering fluids; and additional metal compounds, in particular 1- to 6-valent metal compounds, for example compounds of Na, Ag, Al, Co, Ni, Fe, Ga, In, lanthanides, Zr; Sc, Ti, V, Cr, Mn, Cu, Zn, Y, Nb, Mo, Hf, Ta, W; and anions, in particular halide ions, in particular chloride ions; sulfurous ions, in particular sulfate ions, nitrate ions; phosphoric ions, in particular phosphate ions, diphosphate ions, linear and/or cyclic oligophosphate ions, linear and/or cyclic polyphosphate ions, hydrogen phosphate ions; carboxylic acid anions; and silicon-containing anions, in particular silicate anions; and polymers, corrosion inhibitors; silicic acids, in particular colloidal or disperse silicic acids; surfactants; diols, triols, polyols; organic acids, in particular monocarboxylic acids; amines; plastics dispersions; dyes, pigments, in particular carbon black, chromogenic agents, in particular metallic chromogenic agents; amino acids, in particular glycine; siccatives, in particular cobalt siccative; dispersing agents; as well as mixtures thereof.
19. (amended) A passivation bath according to claim 14, characterised in that it has a bath temperature of approx. 20 to 100°C, preferably 20 to 80°C, in a preferred manner 30 to 60°C, in a particularly preferred manner 40 to 60°C.
20. (amended) A method for passivating surfaces of zinc or zinc alloys, in particular ones with iron,  
**characterised in that** the objects to be treated are immersed in a passivation bath according to claim 14.

22. (amended) A method according to claim 20,  
characterised in that it is an elevated-temperature chromate  
coating method with rinsing water recycling over at least 2  
cascaded rinsing stages.

24. (amended) A passive layer obtainable by a method according to  
claim 20.

26. (amended) A passive layer according to claim 24 , characterised in  
that it presents a greenish, red-green iridescent color for zinc.

27. (amended) A passive layer according to claim 24,  
characterised in that its layer thickness is approx. 100 nm.

28. (amended) A conversion layer obtainable by a method according to  
claim 7.

Please add new claims 29-36 as follows.

29. (new) A conversion layer comprising chromium(III), said conversion layer being chromium(VI)-free, said conversion layer being a substantially coherent conversion layer on zinc or a zinc alloy, said conversion layer presenting a corrosion protection of about 100 to 1000 h in the salt spray test according to DIN 50021 SS or ASTM B 117-73 until first attack according to DIN 50961 Chapter 10, said conversion layer having an average chromium content of more than approximately 5% based on zinc and chromium, said conversion layer having a chromium index greater than approximately 10, wherein the chromium index is defined as said average chromium content ( $\text{chromium}/(\text{chromium} + \text{zinc})$ ) in the layer greater than 1% Cr, multiplied by the layer thickness in nm.

30. (new) A conversion layer according to claim 1, said layer being free from the presence of silicate, cerium, aluminum, and borate.

31. (new) A conversion layer according to claim 1, said layer having a layer thickness of about 100 nm to 1000 nm, said conversion layer having across the conversion layer thickness a chromium content of greater than 1% based upon zinc and chromium.

32. (new) A conversion layer according to claim 1, said layer being clear or substantially colorless.

33. (new) A conversion layer according to claim 1, said layer further comprising cobalt.

34. (new) A conversion layer according to claim 1, said layer further comprising one or more metal compounds selected from the group consisting of 1- to 6-valent metal compounds.

35. (new) A conversion layer according to claim 1, said

layer further comprising one or more anions.

36. (new) A conversion layer according to claim 29, said layer further comprising one or more materials selected from the group consisting of polymers, corrosion inhibitors, silicic acids, surfactants, polyols, organic acids, amines, plastics, dispersions, dyes, pigments, chromogenic agents, amino acids, siccatives, dispersing agents, organic polymers, diols, triols, monocarboxylic acids, carbon black, metal chromogenic agents, glycin, and cobalt siccatives.

REMARKS

The claims filed in the parent application have been amended and new claims have been added for prosecution. No new matter is introduced into the application by the new claims. Also the cross-reference to the parent application has been added to the specification.

If any fees are required by this communication which are not covered by an enclosed check, please charge such fees to our Deposit Account No. 16-0820, Order No. 31716US1.

Respectfully submitted,

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Date: 2-13-01

3. A conversion layer according to claim 1[er 2], characterised in that it may contain, for further enhanced corrosion protection, additional components selected from the group consisting of: silicate, cerium, aluminum and borate;

additional metal compounds, in particular 1- to 6-valent metal compounds, for example compounds of Na, Ag, Al, Co, Ni, Fe, Ga, In, lanthanides, Zr; Sc, Ti, V, Cr, Mn, Cu, Zn, Y, Nb, Mo, Hf, Ta, W; and

anions, in particular halide ions, in particular chloride ions; sulfurous ions, in particular sulfate ions, nitrate ions; phosphoric ions, in particular phosphate ions, diphosphate ions, linear and/or cyclic oligophosphate ions, linear and/or cyclic polyphosphate ions, hydrogen phosphate ions; carboxylic acid anions; and silicon-containing anions, in particular silicate anions; and

polymers, in particular organic polymers, corrosion inhibitors; silicic acids, in particular colloidal or disperse silicic acids; surfactants; diols, triols, polyols; organic acids, in particular monocarboxylic acids; amines; plastics dispersions; dyes, pigments, in particular carbon black, chromogenic agents, in particular metallic chromogenic agents; amino acids, in particular glycine; siccatives, in particular cobalt siccatives; dispersing agents; and

mixtures thereof.

4. A conversion layer according to ~~any one of~~ claims 1[er 2] characterised in that it is a basis for further inorganic and/or organic layers.

5. A conversion layer according to ~~any one of~~ claims 1[er 2] characterised in that it contains dyes or color pigments for modification of the inherent color thereof.

6. A conversion layer according to ~~any one of~~ claims 1[er 5] characterised in that its layer thickness is approx. 100 nm.

9. A method according to [any one of] claims [7 or 8] characterised in that the ligands of the chromium(III) complex are selected from the group consisting of:

chelate ligands, such as dicarboxylic acids, tricarboxylic acids, hydroxycarboxylic acids, in particular oxalic, malonic, succinic, glutaric, adipic, pimelic, suberic, azelaic, sebacic acid; and

furthermore, maleic acid, phthalic acid, terephthalic acid, tartaric acid, citric acid, malic acid, ascorbic acid; and

further chelate ligands such as acetylacetone, urea, urea derivatives, and

further complex ligands wherein the complexing functional group contains nitrogen, phosphorus or sulfur (-NR<sub>2</sub>, -PR<sub>2</sub>, wherein R independently is an organic, in particular aliphatic radical and/or H, and/or -SR, wherein R is an organic, in particular aliphatic radical or H); phosphinates and phosphinate derivatives; as well as

suitable mixtures thereof, among each other as well as in mixed complexes with inorganic anions and H<sub>2</sub>O and/or

the method is performed repeatedly on the surface to be passivated.

12. A concentrate according to [any one of] claims [10 or 11] characterised in that the concentrate is present in solid or liquid form.

13. A concentrate according to [any one of] claims [10 to 12] characterised in that it contains further additives selected from the group consisting of: sealers, dewatering fluids; and additional metal compounds, in particular 1- to 6-valent metal compounds, for example compounds of Na, Ag, Al, Co, Ni, Fe, Ga, In, lanthanides, Zr; Sc, Ti, V, Cr, Mn, Cu, Zn, Y, Nb, Mo, Hf, Ta, W; and

anions, in particular halide ions, in particular chloride ions; sulfurous ions, in particular sulfate ions, nitrate ions; phosphoric ions, in particular phosphate ions, diphosphate ions, linear and/or cyclic oligophosphate ions, linear and/or cyclic polyphosphate ions, hydrogen phosphate ions; carboxylic acid anions; and silicon-containing anions, in particular silicate anions; and

polymers, in particular organic polymers, corrosion inhibitors; silicic acids, in particular colloidal or disperse silicic acids; surfactants; diols, triols, polyols; organic acids, in particular monocarboxylic acids; amines; plastics dispersions; dyes, pigments, in particular carbon black, chromogenic agents, in particular metallic chromogenic agents; amino acids, in particular glycine; siccatives, in particular cobalt siccatives; dispersing agents; as well as

mixtures thereof.

16. A passivation bath according to claim 14~~or 15~~, characterised in that it has a pH between approx. 1.5 and 3.
17. A passivation bath according to ~~any one of~~ claims 14~~to 16~~, characterised in that it contains approx. 20 g/l chromium(III) and has a pH of approx. 2 to 2.5.
18. A passivation bath according to ~~any one of~~ claims 14~~to 17~~, characterised in that it contains further additives in particular selected from the group consisting of sealers, dewatering fluids; and additional metal compounds, in particular 1- to 6-valent metal compounds, for example compounds of Na, Ag, Al, Co, Ni, Fe, Ga, In, lanthanides, Zr; Sc, Ti, V, Cr, Mn, Cu, Zn, Y, Nb, Mo, Hf, Ta, W; and

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anions, in particular halide ions, in particular chloride ions; sulfurous ions, in particular sulfate ions, nitrate ions; phosphoric ions, in particular phosphate ions, diphosphate ions, linear and/or cyclic oligophosphate ions, linear and/or cyclic polyphosphate ions, hydrogen phosphate ions; carboxylic acid anions; and silicon-containing anions, in particular silicate anions; and

polymers, corrosion inhibitors; silicic acids, in particular colloidal or disperse silicic acids; surfactants; diols, triols, polyols; organic acids, in particular monocarboxylic acids; amines; plastics dispersions; dyes, pigments, in particular carbon black, chromogenic agents, in particular metallic chromogenic agents; amino acids, in particular glycine; siccatives, in particular cobalt siccative; dispersing agents; as well as

mixtures thereof.

19. A passivation bath according to any one of claims 14 to 18, characterised in that it has a bath temperature of approx. 20 to 100°C, preferably 20 to 80°C, in a preferred manner 30 to 60°C, in a particularly preferred manner 40 to 60°C.
20. A method for passivating surfaces of zinc or zinc alloys, in particular ones with iron,

**characterised in that**

the objects to be treated are immersed in a passivation bath according to any one of claims 14 to 19.

22. A method according to any one of claims 20 to 27, characterised in that it is an elevated-temperature chromate coating method with rinsing water recycling over at least 2 cascaded rinsing stages.
24. A passive layer obtainable by a method according to at least one of claims 20 to 23.

26. A passive layer according to claim 24~~or 25~~, characterised in that it presents a greenish, red-green iridescent color for zinc.
27. A passive layer according to ~~any one of~~ claims 24~~to 26~~, characterised in that its layer thickness is approx. 100 nm.
28. A conversion layer obtainable by a method according to ~~at least one of~~ claims 7~~to 9~~.